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(54) Name of the invention: Alumina-Zirconia-Titania Type Grinding Material

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(71) Patent Assignee: Nippon Kenmazai Kogyo KK

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# (54) Alumina-Zirconia-Titania Type Grinding Material

# Detailed Description of the Invention

### 1. Name of the Invention

# Alumina-Zirconia-Titania Type Grinding Material

# 2. Scope of the Claims of the Invention

- (1) Alumina-zirconia-titanium type grinding material that is a material obtained as zirconia and titania are added to alumina and melted, and then rapidly cooled, where then as another melting additive material yttrium oxide or rare earth type mineral ore material containing yttrium oxide are included.
- (2) Alumina-zirconia-titanium type grinding material according to Claim 1 of the claims of the present invention where the added amount of yttrium oxide is in the range from 0.05 weight % to 7 weight %, relative to the total amount of the alumina, zirconia, and titania.
- (3) Alumina-zirconia-titanium type grinding material according to Claim 1 of the claims of the present invention where the added amount of the yttrium oxide containing rare earth type ore material is in the range from 0.05 weight % to 7 weight %, relative to the total amount of the alumina, zirconia, and titania.

# 3. Detailed Explanation of the Invention

This invention is an invention about the improvement of the grinding (polishing) performance of alumina-zirconia-titania type grinding material.

Usually, the alumina-zirconia-type polishing particles are polishing particles that have been quickly expanded in their use as "snacking" of the steel type materials like specialty or stainless steel materials. Namely, compared to the alumina type polishing particles, they have excellent wear resistance and together with that breakage resistance etc., polishing properties, and the fact that under high pressure conditions, they demonstrate superior polishing strength, is the main factor. According to the invention reported in the description of the Japanese Patent Report Number Showa 48-35594, that has been invented previously by the authors of the present invention, an alumina-zirconia-titania material has been obtained that has a polishing performance that is even further increased and more superior than that.

Namely, it is a material where zirconia is added to alumina and together with that then titanium oxide is added in an amount that is in the range of  $5 \sim 30$  weight % relative to this zirconia amount, and then melted, and because of the fact that it is then quickly cooled, the phenomenon of the phase transformation back to the original zirconia is suppressed, and because of that the high temperature type tetragonal system crystalline phase remains present in an amount in the range of  $25 \sim 30$  % at normal temperatures, and due to that, it contributes to the increase in the wear resistance properties, breakage resistance properties, etc., polishing properties performance.

However, recently, there is a trend where the polishing methods have also constantly been changing, and the application of fine materials in the so-called heavy grinding field, and especially among those, the significant development of the technology of the change to the light grinding field within the heavy grinding, and also the use of the polishing materials for polishing relative to titanium alloys, has increased. A grinding material that is appropriate for this field is currently required, however, in this case, the required grinding performance is a performance that is higher than that of the above described alumina-zirconia-titania type grinding material, and especially, it is a material that cannot be a severe type of material, and the current status is that a grinding material with satisfactory grinding properties has not yet been obtained.

Namely, in order to be appropriate for the above-described application, it is necessary to suggest a grinding material where however it is stated it has a relatively high amount of zirconia, for example, a grinding material that is formed from a alumina-zirconia co-crystalline material of the type where the amount of zirconia is 40 %. However, in this case, even though it is an

alumina-zirconia-titania material obtained through the above-described titania addition, the ratio of the remaining existing high temperature type tetragonal crystal is extremely low, and the result from the addition is poor. Also, there have been the problems that the initial crystalline alpha-Al2O3, whose crystal size in the mixed crystalline material is a maximum of 20 microns, is separated at a level of  $10 \sim 15$  %, and because of that, the predetermined co-crystalline mixed material could not be obtained.

Then the goal of the present invention is to suggest an alumina-zirconiatitania type grinding material where in order that this material be good for application especially relative to heavy grinding, and especially to the light grinding, and then also, relative to titanium alloys, the remaining present amount of the tetragonal crystalline material is extraordinarily increased, and also, the coefficient of the separation of the original crystals of the alpha-Al2O3 is reduced, and because of that the grinding performance is significantly increased. And the characteristics of this material are the fact that it is an alumina-zirconia-titania type grinding material, where then as another additive material yttrium oxide or yttrium oxide containing rare earth ore material, is contained, and it is then called an alumina-zirconia-titania-yttria type grinding material.

Namely, as the additive materials, besides titania, yttrium oxide or yttrium oxide containing rare earth ore material are then added and mixed, and melted, and quickly cooled and by that, a grinding material is obtained that achieves well and satisfies the above described goal. And especially, it is a material where the added amount of the yttrium oxide or yttrium oxide containing rare earth ore material is within the range where it exceeds 0.5 weight % and is up to 7 weight % relative to the total amount of the alumina, zirconia, and titania, and where the zirconia tetragonal crystalline material is crystal separated at a remaining ratio that is in the range of  $70 \sim 100$  %. And not only that but also, within the same range, the ratio of the separation of the initial crystalline alpha-Al2O3 can be suppressed to a level below 5 weight %.

Moreover, even in the case when the added amount is higher than 7 weight % or below 0.05 weight %, the proportion of the remaining zirconia tetragonal crystal material shows a vale that is relatively high compared to the previous technology, and also, the ratio of the separated initial alpha-Al2O3 has a trend where it is significantly suppressed. However, in the case where the added amount exceeds 7 weight %, this leads to the separation of

a cubic crystalline phase, which is not preferable from the point of view of the grinding performance. And in the case when the added amount s less than 0.05 weight %, significant results are not obtained and because of that the range where the added amount is higher than 0.05 weight % and it is up to 7 weight %, is most appropriate, and especially, it is most appropriate that the added amount is within the range of  $1 \sim 5$  weight %.

Moreover, even, relative to a grinding material that contains an alumina-zirconia co-crystalline material with a relatively small amount of zirconia, for example, 25 %, the same way as in the case of the above-described grinding material that is formed from an alumina-zirconia co-crystallized material that contains a relatively large amount of zirconia, the ratio of the remaining tetragonal crystalline phase zirconia is extremely high, and because of that, the volume change, which occurs at the zirconia transition point is small, and also, the remaining present tetragonal crystalline type zirconia maintains sufficiently its inner part energy, and it has been concluded that there is a trend of improvement of the properties, like good breakage resistance properties, etc.

After that, the practical implementation examples according to the present invention will be explained.

Moreover, as the yttrium oxide or yttrium oxide containing rare earth ore material, that are used in the practical implementation examples according to the present invention, the materials with the analyses values shown according to Table 1, were used.

Table 1

1 31 38 2						
	<b></b>					
	7   酸化イットリウム	が 酸化イットリウムを含む				
	(wt%)	据土斑鉱物 (wi%)				
Y2 O3	99.9	60				
l·e z Os	0,0015	0.013				
Na z O	0.0015	-				
K1 0	0,0015	-				
Si Oz	0,001	0.05				
l-a 5 O3	-	< 10				
Ce Oz	_	<10				
Nd 2 O3		< 10				
Yb 2 O3	_	6				
Er 2 Oı	-	6,5				
SR 2 O1	_	2				

Headings in the table:

1. yttrium oxide (weight %), 2. rare earth ore material containing yttrium oxide (weight %).

Table 2:

	·l			ফা 2	돲			
500 番页	Y2 O		710	Dz Ti O	Y 2 C	Feac	Si C	De Na z O
1	0	50,94	41.61	1.20	_	0.05	0, 12	0,08
2	0.05	57.43	41.25	1.00	0.03	0,04	0.13	0.03
3	0.1	57.91	40,75	1,05	1,06	0.05	0, 14	0.04
1	0.5	57.65	40,85	1.02	0.27	0.03	0.14	. 0.04
5	1	57, 12	40.88	1,10	0,64	0,06	0.16	0.04
G	2.5	55,92	41.30	1.03	1.52	0.05	0.14	0,04
1	5	55, 16	40,45	1.05	3.00	0,04	0.18	0.03
0	10	52.47	40,12	1.11	6.05	0.05	0, 17	0.03

Headings in Table 2:

1. Material number, 2. Y2O3 added amount (weight %).

# Practical Example 1

A compounding calculation was conducted so that the zirconia in the grinding particles became 40 %, and to 50 kg of Bayer method alumina (99.6 % Al2O3), 32.6 kg zirconia (96 % Zr02), 0.8 kg of titanium oxide (95 % TiO2) which corresponds to 2.5 in weight % relative to the zirconia, were mixed and combined, and then, yttrium oxide (99.9 % Y2O3) was added in amount of 0.05, 0.1, 0.5, 1, 2.5, 5, 10 weight % relative to the total amount of the above described three types of materials, and this was then melted using an electrical oven at 95 V, 300 kV, and after that, it was quickly solidified, and melt cast materials were obtained for each addition amount.

Moreover, for the sake of a comparison, a melt cast material where the addition amount of the yttrium oxide was 0 weight %, was also cast.

The values obtained from the analysis of this cast materials, are shown according to the presented in table 2.

After that, the obtained cast material was repeatedly crushed by using an impeller breaker and a crusher, and particle sizes of # 24, and # 60, as determined according to the procedures of the JISR-6001, were withdrawn and obtained.

The ratio of the zirconia crystalline phases of the particle material group # 24, determined by X-Ray Diffraction analysis, is shown according to the presented in Table 3.

Table 3:

1	2	3 4 3	4
此四百月	/ 正方瓜Zr O2 (%)	<b>単四級ZrOz (%)</b>	プ 立方品Zr O1 (%)
1	30.1	69.9	0
2	32.1	67.9	0
3	70,7	29,3	0
4	87.9	12,1	0
5	100	0	0
6	100	0	0
7	100	0	. 0
8	85,8	0	14, 2

### Headings in table 3:

1. Experimental Material number, 2. tetragonal Zr02 (%), 3. monoclinal crystal phase ZrO2 (%), 4. cubic crystal phase ZrO2 (%).

As it is clear from the results of the X-Ray Diffraction Analysis, if yttrium oxide is added, compared to the alumina-zirconia-titania type grinding particles according to the previous technology where there is no such addition (Experimental material number 1), it is concluded that there is a trend of a significant increase of the tetragonal crystalline phase amount. And especially, it was confirmed that by the addition of an additive amount in the range of 1 ~ 5 weight %, there was a 100 % tetragonal crystal phase

crystal separation. Also, in the case of the addition of 0.05 weight %, there was a corresponding trend of increase of the amount of the separated tetragonal crystal phase, however, there was no significant difference with the previous technology material, and also, if the added amount exceeded 10 weight %, a separation of a cubic crystalline phase was observed.

On the other hand, at the time when the separation of the initial alpha-Al2O3 of for example a material according to the previous technology (Experimental material 1) and material where the added amount of the yttrium oxide was 0.5 weight % (Experimental material 3), with the same # 24 particle size, was observed by using a metal observation microscope, the results that are shown according to the presented in Figure 1 (A), (B) and Figure 2 (A), (B), were obtained. In each case, the magnification was x 100, and Figure 1 (A), (B), represents an enlarged microphotograph of the Experimental material 1, and its model diagram, and Figure 2 (A), (B), represents an enlarged microphotograph of the Experimental material 3, and its model diagram.

Moreover, in both figures, 1 represents the alumina-zirconia co-crystalline material, and 2 - the alpha-Al2O3 initial crystals.

As it is clear from Figure 1 and Figure 2, in the case of the material with the addition of the yttrium oxide, the separation of the initial alpha-Al2O3 crystals was significantly suppressed, and it was confirmed that the predetermined co-crystalline mixed material, was obtained.

Moreover, it was confirmed that the same trend exits in the case of the other materials where yttrium oxide has been added, however, in the case when the added amount was 0.05 weight %, the separated alpha-Al2O3 was at a level of 10 %, and because of the fact that the material according to the previous technology had a level in the range of  $10 \sim 15$  %, this trend was almost negligible.

After that, grinding performance tests were conducted relative to the materials with the particle size of # 60.

Namely, a polishing belt was prepared and a polishing test was conducted and the results from that test are shown according to the presented in Table 4.

Moreover, the polishing test was conducted as the size of the belt used was 100 x 2500 m/m, and as the material subject to the polishing SUS-304 was used, and the belt speed was set at 150 m/minute, and the pressure was at 5 kg, and a 10 minute polishing was conducted. Table 4 is a table that shows the comparison of the cumulative mass polished amounts that were obtained by this polishing.

Also, the number in the parentheses shows the comparative value when the case of the material obtained according to the previous technology (Experimental material 1) is set as 100.

Table 4:

	11 1 3k
<b>以知前用</b>	深格明初后 (g )
1	94.4 (Ralin 100)
2	95.0 ( * 100)
J	113.0 ( # 120)
4	115.7 ( # 123)
5	110.2 ( # 126)
. 6	120.6 ( * 128)
1.7	120.4 ( # 127)
8	96.0 ( * 101)

Headings in the table:

1. Experimental material number, 2. cumulative mass polished amount (g).

Also, the curve line (a) in Figure 3 represents a graph where the cumulative mass polished amounts presented in Table 4, have been plotted.

As it is clear from the above described Table 4 and Figure 3, it has been confirmed that in the case of the co-crystalline grinding particles where yttrium oxide has been added, an extremely excellent polishing strength has been demonstrated compared to the material according to the previous technology where yttrium oxide has not been added.

In this connection, these results are results that correspond to the ratios of the zirconia tetragonal crystalline lattice crystal material, and in the case of the material where the addition amount was 0.05 weight %, there was no significant difference with the material according to the previous technology; and also, in the case when the added amount exceeded 10

weight %, a trend down from the material according to the previous technology was observed due the accompanying separation of cubic crystalline phase material.

However, the addition of the yttrium oxide significantly increases the polishing strength as a total, and not only that, but also, when it is considered that this test results represent only a comparison at the time when a 10 minute polishing has been completed, it is clear that a material is obtained which during the use in practice contributes to the improvement and to achieving extremely excellent polishing performance.

# Practical Example 2

A compounding calculation was conducted so that the zirconia in the grinding particles became 40 %, and to 50 kg of Bayer method alumina (99.6 % Al2O3), 32.6 kg zirconia (96 % Zr02), 0.8 kg of titanium oxide (95 % TiO2) which corresponds to 2.5 in weight % relative to the zirconia, were mixed and combined, and then, three types of yttrium oxide containing rare earth mineral ore (the materials with analyses values shown according to the presented in Table 1) was added in an amount of 1, 2.5, 5 weight % relative to the total amount of the above described three types of materials(zirconia, alumina, titania), and this material was then melted using an electrical oven at 95 V, 300 kV, and after that, it was quickly solidified, and melt cast materials were obtained for each addition amount.

The values obtained from the analysis of this cast materials, are shown according to the presented in table 5. Moreover, the experimental material number 1 is the same as that in the case of the Practical Example 1 and it is a material according to the previous technology where yttrium oxide has not been added.

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सम बाह्य	Y2 O) を / 含むは上類 はなの既加別	Al <sub>2</sub> O <sub>2</sub>	Zr Oz	TI Oz	मः	F0403	Si O	Na z O
,		56,94	41,61	1.20		0.00	0, 12	0.08
3	1	57.36	40.97	1,04	0.42	0.05	0, 13	0,03
10	2.5	56, 56	41.04	1.09	1.05	0.06	0, 16	0.04
11	5	56, 19	10,33	1,12	2.11	0.05	0,17	0.03

### Headings in Table 5:

1. Experimental material Number, 2. Added amount of yttrium oxide containing rare earth mineral ore (weight %).

After that, the obtained cast material was repeatedly crushed by using an impeller breaker and a crusher, and particle sizes of # 24, and # 60, as determined according to the procedures of the JISR-6001, were withdrawn and obtained.

The ratio of the zirconia crystalline phases of the particle material group # 24, determined by X-Ray Diffraction analysis, is shown according to the presented in Table 6.

Table 6:

1	2	n 6 ts 3	4
क्षानम् म	  正方型Z r Oz (%)	即即及Zr Ot(%)	Setslin Zr Oz (%)
1	30,1	69,9	0
9	96, 4	3, 6	. 0
10	100.0	0	0
11	100.0	. 0	n

### Headings in table 6:

1. Experimental Material number, 2. tetragonal Zr02 (%), 3. monoclinal crystal phase ZrO2 (%), 4. cubic crystal phase ZrO2 (%).

As it is clear from the results of the X-Ray Diffraction Analysis, if yttrium oxide containing rare earth mineral ore is added, the same way as in the case of the addition of yttrium oxide, compared to the alumina-zirconia-titania type grinding particles according to the previous technology where there is no such addition (Experimental material number 1), it is concluded that there is a trend of a significant increase of the tetragonal crystalline phase amount.

<sup>\*</sup>Rare earth type other than Y2O3.

And it was confirmed that by the addition of an additive amount in the range of  $2.5 \sim 5$  weight %, there was a 100 % tetragonal crystal phase crystal separation.

After that, grinding performance tests were conducted relative to the materials with the particle size of # 60.

The experimental conditions are the same as those in the case of the Practical Example 1 and the results from that test are shown according to the presented in Table 7.

### Table 7:

1	第7表 、 シ
試取新号	深松期前出(g)
1	94.4 (Ratio 100)
9	118.7 ( * 126)
10	120.0 ( - 127)
11	119.2 ( ~ 126)

Headings in the table:

2. Experimental material number, 2. cumulative mass polished amount (g).

Also, the curve line (b) in Figure 3 represents a graph where the cumulative mass polished amounts presented in Table 7, have been plotted.

As it is clear from the above described Table 7 or Figure 3, it has been confirmed that in the case of the co-crystalline grinding particles where yttrium oxide containing rare earth mineral ore has been added, the same way as in the case described in the Practical Example 1 where yttrium oxide was added, an extremely excellent polishing strength has been demonstrated for the obtained co-crystalline grinding particles.

# Practical Example 3

A compounding calculation was conducted so that the zirconia in the grinding particles became 40 %, and to 50 kg of Bayer method alumina (99.6 % Al2O3), and to 32.6 kg zirconia (96 % Zr02), 1.7 and 4.9 kg of titanium oxide, which corresponds to 5 and 15 weight % relative to the zirconia, were mixed and combined, and then, yttrium oxide was added in an

amount of 0.5 weight % relative to the total amount of the above described three types of materials. And for this material, the results from the analyses of the cast material, the ratio of the zirconia crystalline lattice types according to the X-Ray Diffraction for the particle size # 24, and the cumulative mass polished amount for the particle size # 60, were measured.

These results are correspondingly shown according to the presented in Table 8, Table 9 and Table 10.

Moreover, for the sake of a comparison, the values in the case when there was no addition of yttrium oxide regarding the material where 2.5 weight % of titanium oxide was added, and the materials with the corresponding titania addition amounts, are also reported.

Also, in each case the testing methods are the same as those reported according to the Practical Example 1 and the Practical Example 2.

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	Γя	h	le	8	•

l	01	3		ង្វា (	B &				*****
36.00 8.03	TiO. SSIOD (%)	(M)%)	A 1.05			Ya Os	راره۶	SI O <sub>4</sub>	Na_0
i	2,5	0	56.94	41,61	1,20	1	0,05	0.12	0,06
4	2,5	0.5	57,65	40,85	1.02	0.27	0,03	0, 14	0.04
12	5	0	57.06	40,63	2,06	~	0,03	0, 18	0,04
13	5	0,5	56, 78	40,71	2,04	0.25	0.01	0, 15	0,03
14	15	0	54, IG	39,72	5,91		٥,03	0, 14	0.04
15	15	0.5	53, 41	40,24	S,A3	0.29	0.04	0. 15	0.04

Headings in table 8:

1. Experimental material number, 2. TiO2 added amount (%), 3. Y2)3 added amount (%).

Table 9:

,\	2 #98	.3
試料指房	正方品之口口:	HERZ TO:
	(%)	(%)
1	30.1	69,9
4	47,9	12.1
12	33.0	67.0
13	90.2	9.8
14	34.2	65,8
15	93.6	G . 4

Headings in Table 9:

1. Experimental Material number, 2. tetragonal Zr02 (%), 3. monoclinal crystal phase ZrO2 (%).

Table 10:

1	新10数 2			
試和報用	72 Ki	~ GR	相可	( n )
1	. 94.4	(	Rai	io 100)
4	115.7	(	*	123)
12	95.0	•	•	100)
13	,112,5	(		119)
1 4	92.0	ţ	•	97)
1.5	117.3	(		124)

Headings in Table 10:

1. Experimental material number, 2. cumulative mass polished amount (g).

As it is clear from the tables, it is shown that together with the increase of the addition amount of the titanium oxide, there is a trend of increase of the ratio of the remaining present tetragonal crystalline phase Zr02, however, to that, also, by the addition of the yttrium oxide, a significant increase trend was observed.

# Practical Example 4

The materials obtained as compounding calculations were conducted so that the zirconia in the grinding particles became 25 % and 32 %, and relative to 50 kg and together with that 24.5 kg of Bayer method alumina (99.6 % Al2O3), and 16.6 kg and together with that 11.5 kg of zirconia (96 % ZrO2), correspondingly as weight percent, 0, 2.5, 5, 10, 15, 20, 30 and 40 % of titanium oxide (95 % TiO2) were added, and the materials where compounding calculations were conducted so that the zirconia in the grinding particles became 25 %, and where relative to 50 kg of Bayer method alumina (99.6 % Al2O3); and 16.6 kg of zirconia (96 % ZrO2), correspondingly as weight percent, 0, 5, 10, 15, 20, 30 and 40 % of titanium oxide (95 % TiO2) were added, and then yttrium oxide was correspondingly added in an amount of 0.5 weight % relative to the total amount of the above described three types of materials, were correspondingly then melted by an arc heat using an electrical oven, and after that, it was quickly solidified, and these materials were then made into particles by following the usual grinding

particle manufacturing methods, and they were made into JIS #12 grinding particles.

These materials, namely, relative to the types without the yttrium oxide addition, which contain 25 % zirconia and 32 % zirconia, and the type where yttrium oxides has been added and which contains 25 % zirconia, the break resistance measurement test was conducted. For the break resistance measurement test the single particle break strength was used. In this method, the experimental material is granulized to a particle size in the range of 1680 ~ 2000 microns, and by the shrinkage method it is made into a small experimental material, and from within that sample, randomly, 100 units are taken out, and these are measured for their pressure resistance strength one by one, by using a 2 ton amsura compressing equipment. The average value of these measurements was used as the value for the single particle break pressure strength.

The single particle break pressure strength of these grinding particles is shown according to the presented in Table 11 and the relationship between the single particle break pressure strength and the TiO2/ZrO2 ratio, is presented in Figure 4.

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•	uo,		_	T	•

. (	2	J	<b>1</b> 0 1	1 &					
KH 都身	9 1 7			TI	02 /	Zr O	: (%	)	
-		0	2,5	3	10	15	20	30	40
10	Y: O: 無抵用 Zr O: 25% タイプ ク	ľ	48.8	51,8	61.0	60.0	58,9	52.2	4G,7
17	Y2 O2 和級加 Zr O2 12% タイプ 〜	46.3	47,5	53,8	63.0	63.3	62.0	49,3	45.0
18	Y: O: 加延加 Zr O: 25% タイプ	45, 1	-	55,8	04.7	64.2	63.3	54,1	48,0

### Headings in Table 11:

1. Experimental material number, 2. type, 3. type without addition of Y2O3, and 25 % ZrO2, 4. type without addition of Y2O3 and with 32 % ZrO2, 5. type with the addition of Y2O3 and with 25 % ZrO2.

As it is clear from the presented in Figure 4, in the case of any of these types of materials, when the ratio TiO2/ZrO2, expressed as weight %, is within the range of  $10 \sim 20$  %, the strength shows a maximum value, and when the ratio becomes 40 %, there is a decrease. And especially, in the case of the material where yttrium oxide has been added, it was confirmed, naturally, that the type of material where the amount of the ZrO2 was 25 % is a material which demonstrates an especially excellent break resistance performance compared that in the case when 32 % of zirconia has been added.

As described here above, in the case of the present invention, it is an invention about an alumina-zirconia-titania type grinding material, that is formed as to alumina, zirconia and titania are added and melted, and then quickly cooled, and then, as another melt additive material, yttrium oxide or yttrium oxide containing rare earth mineral ore is contained, and by that, it becomes a material where the maximum remaining zirconia tetragonal crystalline phase is 100 %, and also, even though it is a grinding material that is formed from an alumina-zirconia co-crystalline material with a relatively high amount of zirconia, the amount of the separated (precipitated) initial crystals of the alpha-Al2O3 is suppressed to less than 5 %, and because of that it is possible to suggest a material which has a significantly improved grinding performance.

Consequently, it is a material that can be used appropriately in heavy grinding also, and especially, in its light grinding field, and also then, it can be appropriately used relative to titanium alloys.

Also, even though it is an alumina-zirconia type grinding material that contains a relatively small amount of zirconia, compared to the materials according to the previous technology, it is a material whose break resistance strength etc., properties are further improved.

## 4. Brief Explanation of the Figures

Figure 1 (A), (B), represent the enlarged microphotograph and the model type diagram of the obtained according to the previous technology alumina-zirconia co-crystalline mixed material, which has no added yttrium oxide (Experimental material number 1).

Figure 2 (A), (B), represent the enlarged microphotograph and the model type diagram of the obtained according to one practical example of the present invention alumina-zirconia co-crystalline mixed material, which has added yttrium oxide (Experimental material number 3).

Figure 3 is a diagram showing the relationship between the added amount of yttrium oxide etc., which is found in the grinding material with added yttrium oxide and added yttrium oxide containing rare earth mineral ore, according to the same practical example and another practical example, and the cumulative mass polished amount.

Figure 4 is a diagram showing the relationship between the added amount of the titanium oxide found in the grinding material with relatively low zirconia amount and the single particle pressure break strength.

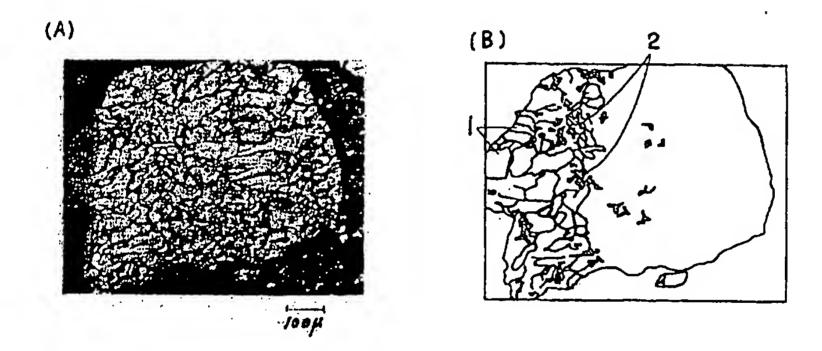


Figure 1: (A) and (B)

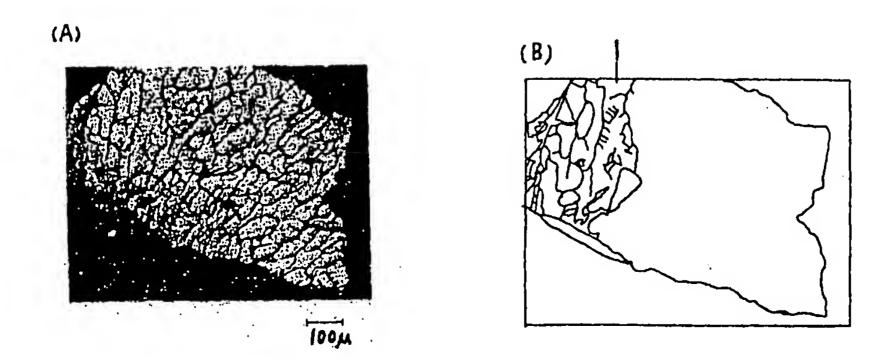
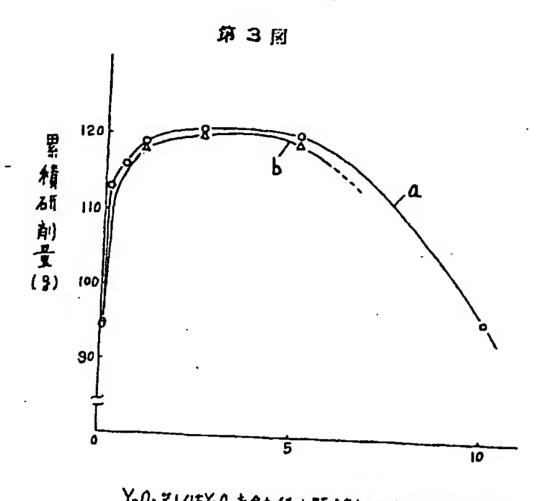


Figure 2: (A) and (B)



Y20,若LCIFY20,老含む稀土類鉱物の添加量(wt%)

(a)..... Y20101110

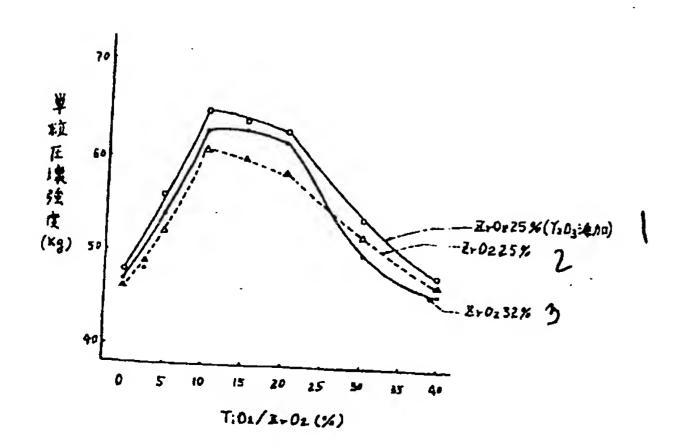
(b)……2012含6稀土類鉱物の添加

Figure 3:

On the vertical axis – cumulative mass polished amount (g)
On the horizontal axis – added amount of Y2O3 or Y2O3 containing rare
earth mineral ore (weight %)

(a).....Y2O3 added amount

第4 图



### Figure 4:

On the vertical axis – single particle pressure break strength (kg) On the horizontal axis – TiO2/ZrO2 (%)

- 1. ZrO2 25 % (no Y2O3 added)
- 2. ZrO2 25%
- 3. ZrO2 32 %

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①特許出願公開

### ⑩公開特許公報(A)

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**匈**アルミナージルコニアーチタニア系研削材

②特

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明期世

1. 発明の名称

アルミナージルコニアーチタニア系 研削材

2.特許請求の範囲

(1) アルミナドジルコニア及びチクニアを抵加密 融し、急冷してなるアルミナージルコニアーチタ ニア系研削材において、さらに他の溶験 窓加物と して酸化イットリウム又は酸化イットリウムを含 む稀土類鉱物を含有するアルミナージルコニアー チタニア系研削材。

(2) 酸化イットリウムの添加量が、アルミナ、ジルコニア、及びチタニアの全世に対し O. O 5 Wt %を超え 7 wt %に至る特許請求の範囲第 1 項配収のアルミナージルコニアーチタニア系研削材。

(3) 酸化イットリウムを含む稲土類鉱物の添加量が、アルミナ、ジルコニア及びチタニアの全量に対し〇. 〇 5 W (%を超えて W)%に至る特許請求の範囲第1項記収のアルミナージルコニアーチタニ

ア系研削材。

3. 発明の詳細な説明

この発明はアルミナージルコニアーチタニア系 研削材における研削性能の改良に関するものであ る。

一般にアルミナージルコニア系 砥粒は、ここ数年間において 特殊 網やステンレス 網等の 鉄 楓 粒でのスナッキング用として 急速に 仲 びてき 耐 軽 性 立 なっ かっちアルミナ系 砥粒に 役 れ、高圧 下 で がいて 優 秀 な 研 削 力 を 発 仰 す ること が そ の 主 因 で が な が 、 本 発 明 者 が 先 に 別 示 し た 特 公 昭 4 8 0 0 3 5 5 9 4 月 公 郊 所 較 の 発 明 で は 、 そ れ に も が ル こ か が ら ら に 一 図 す ぐ れ に 研 削 性 能 を 具 備 す る ア ル コ ニ ア ー チ タ ニ ア 系 研 削 材 を 促 供 し 得 た も の で あっ た。

すなわち、アルミナにジルコニアを加えるとともに、そのジルコニア母に対し5~30wt%の酸化チタンをさらに添加して溶融し、急冷するというもので、ジルコニア本来の転移現象に着目し、

#### 特開昭59-227726(2)

高温型正方晶結晶を常温において 25~30%程 度 残存させ、もって耐摩耗性、抗破砕性等の研制 性能の向上に寄与せしめたものであった。

すなわち上記のごとき用途に適応させるためには、どちらかといえばジルコニア量を比較的多量とした例えばジルコニア量40%タイプのアルミナージルコニア共品体からなる研削材を提供する必要があるが、この場合では、前述のチタニア

加によるアルミナージルコニアーチタニア系の間付けであっても高温型正方晶結晶の残存率は未だ極めて低く、 透加効果は乏しいものであり、また結晶の大きさが max 2 0 μもある初島のαーΑ I 2 O 1 が 1 0~15%程度の比率で析出することから、所定の共晶混合物を符られないといった関節点を有していたものである。

でこの狂明の目的とするとは、近の狂明の目的とするとない。 の中でも特している。 の自知のはいる。 の自知のはいいでは、 の自知のはいいでは、 の自己のないでは、 ののはいいでは、 ののはいいでは、 ののはいいでは、 ののは、 

なお知論 7 Wt %以上もしくは 0 . 0 5 wt %以下の添加量であってもシルコニア正方晶結晶の残存 準は従来に比して比較的大きな値を示し、かつ初 晶の α - A | 2 0 1 の折出率を若干抑制する傾向 はあるが、 7 Wt %以上の添加では研別性能の向上 の点から好ましくない立方結晶の析出につながり、 0 . 0 5 Wt %以下では若しい効果は現われないこ とから、O. O.5 Wt %を越えてwt %に至る範則内が適切で、特に 1 ~ 5 Wt %の範囲内が最も好ましい。

なおまた、比較的少量のジルコニア類、例えば25%タイプのアルミナージルコニア共精品があるのでから、上述の比較的のジルコニア共晶体があるのアルミナージルコニアの転移のからがある。 また残存した正方品がいる できない から、また残存した正方品がいる できない から、また残存した正方品がいる ことが 内部エネルギーを十分な有するに至り、良好な抗酸が性等の特性の向上が認められたものである。

次に本発明の実施例について説明する。

なお、本発明の実施例において使用する酸化イットリウム並びに酸化イットリウムを含む稀土類 鉱物は第1表に示した分析値のものを用いた。

#### 特開昭59-227726(3)

郑 1 赛 酸化イットリウム「酸化イットリウムを含む (wt%) 稀土類鉱物 (w1%) Y2 O3 60 99.9 Fe 2 O3 0,0015 0.013 Na 2 O 0.0015 K<sub>2</sub> O 0.0015 Si Oz 0,001 0.05 La 2 O3 <10 Ce O2 < 10 Nd 2 O3 < 10 Yb 2 O3 Er 2 O3 6,5 Sm 2 O3

实施例1.

低粒中のジルコニアが40%となるように配合計算を行ない、バイヤー法アルミナ(99.6% A I z O z ) 5 0 kg 、 ジルコニア(96% 2 r O z ) 3 2 . 6 kg に、ジルコニアに対し追風バーセントで 2 . 5 % に相当する酸化チタン(95 % Ti O z ) 0 . 8 kg を混合し、さらに酸化イットリウム(99.9% Y z O z )を前3種の全量に対し0 . 0 5 、0 . 1、0 . 5、1、2 . 5、5、1 0 wt % 添加して、電気炉において95 V 3 0 0 kw で溶験し、しかる後急冷切化して各種添加量についての溶験鋳造物を得た。

なお比較のために酸化イットリウムの添加量が O wt%であるዋ壁紡造物も同条件で鋳造した。 これらの鋳造物の分析値を第2装に示す。

(以下次頁)

	2		ន	7.2 ≩	Š.			
ECHER !	Yz O3 添加加 (Wt%)	A IzO3	Zr Oz	TI Oz	Y2 O3	, Fe <sub>2</sub> O <sub>3</sub>	Si Oz	Na 2 O
1	0	56,94	41.61	1.20	-	0.05	0.12	0.08
2	0.05	57,43	41.25	1.09	0.03	0.04	0,13	0.03
3	0,1	57,91	40,75	1,05	1,06	0.05	0.14	0.04
4	0.5	57,65	40.85	1,02	0.27	0.03	0, 14	0,04
5	1	57. 12	40.88	1,10	0_64	0.06	0.16	0.04
6	2,5	55, 92	41,30	1.03	1,52	0.05	0, 14	0,04
7	5	55,16	40.45	1.05	3.00	0,04	0,18	0.03
8	10	52.47	40,12	1,11	6,05	0.05	0, 17	0.03

次にこの何られた紡造物をインペラーブレーカー及びロールクラッシャーを用いて繰り返じ粉砕を行ない、JISR-6001に定められた粒度
#24、#60で各々採取した。

粒度#24についてX線回折によるジルコニアの結晶形の比率を第3表に示す。

(以下次頁)

#### 特周昭59-227726(4)

	×	K E	0	Vī	ŋ	粘	栗	Ŋ)	6	か	Ġ	£	う	ĸ		67	化	1	ッ	۲	ŋ
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ジ	л	<i>,</i> ⊐	=	7.	_	チ	9	=	7	系	H	杨	(	試	13	斱	73	1	)	に	比
U	•	īĒ	方	6	Ø.	枵	A	が	G	L	<	II	大	<b>g</b> .	<b>ప</b>	何	ជៀ	に	あ	ð	ت
٤	tš	27	め	5	n		特	ĸ	1	~	5	w t	%	Ø,	跃	ħN	<b>a</b>	で	IJ		
1	0	0	%	ŋ	ĩE	な	æ	枯	æ	15	a	<b>:11</b>	i	τ	٧,	る	ے	Ł	ß	<b>52</b>	<sub>V</sub>
6	ħ	た	4	ŧ	た		ta		が	0		0	5	w t	%	で	t:1	_	瘀	Æ	方
a	の	ß	u	Ø	ğį	大	極	向	녆.	あ	る	ŧ	の	Ø		挺	来	<b>a</b> )	5	٤	あ
ŧ	9	大	茫	lż	Ġ	< ِ		ŧ	tc	1	0	wt	%	æ	超	ħ	ది	٤	众	方	瓜
Ø	₩ſ	货	が	認	め	5,	ħ	k													

一方、この同じ粒度#24のものであって、例えば提来のもの(試料番号1)と酸化イットリウム盔卸量O.5wt%(試料番号3)のものとについて金属顕微鏡を用いて初扇のαーAlzOIの新出状態を観察したところ、第1回(A)(B)及び第2回(A)(B)に示される様な結果が出た。 いずれも倍率は×100で、第1回(A)(b)は試料番号1のものの拡大写真及びその模式図である。

1	2	SR 3 & 3	4
战四番号	正方品Zr Oz (%)	単四周Zr O₂ (%)	/ 立方級Zr O <sub>2</sub> (%)
1	30,1	69.9	0
2	32,1	67.9	. 0
3	70.7	29, 3	0
4	87.9	12.1	. 0
5	100	0.	0
6	100	. 0	0
7	100	0	0
8	85,8		14.2

なお両図において、1 はアルミナージルコニア 共品体、2 はα-A l z O 3 の初品である。

第1回及び第2回からも明らかなごとく、酸化イットリウムを添加したものの方は若しくαームしょの知品の折出が抑制されており、所定の共晶混合物が得られていることが認められた。なお他の酸化イットリウムの添加のものにあっても同様の傾向があることが確かめられているが、添加量が0.05 wt%ではαーAしょ 0』の析出は10%程度あり、従来のもが10~15%程度であることからすると数分その傾向が出ているとはいうものの、あまり若しい差はなかった。

次に粒度#60について研別性能の試験を行なった。

すなわら研歴ペルトを作成し、研削テストを行なったものであり、その結果については第4数に示す。

なお印刷テストは、使用するベルトサイズを 1 0 0 × 2 5 0 0 m / n 、被研削材をSUS -3 0 4 として、ベルトスピード 1 5 0 m / 分、圧 カ 5 kgで、10分型研削を行なったものであり、 第 4 表はその研削によって得られた異様研削量を もって比較したものである。

また括弧内は従来のもの(試料指約1)を 100とした場合の比較値を示す。

n 1 a			
果和	F RPI	朝日 (	g )
94.4	( )	Ratio	100)
95.0	(	•	100)
113.0	•		120)
115.7	(	•	123}
119.2	{	*	126)
120.6	(	•	128)
120.4	(	•	127)
96.0	(	•	101)
	94.4 95.0 113.0 115.7 119.2 120.6 120.4	2 2 2 3 4 4 4 4 4 4 4 4 4 4 4 4 4	果特研制员 ( 94.4 (Ratio 95.0 ( ** 113.0 ( ** 115.7 ( ** 119.2 ( ** 120.6 ( ** 120.4 ( **

また第3回における曲ね(8)は、この第4妻 における保積研削量値をプロットしたグラフであ

#### 特開昭59-227726(5)

**ቆ** "

上記節4衷あるいは第3回から明らかなように、 酸化イットリウムを添加する共島研粒は酸化イットリウムを添加しない従来のものに比して極めて 優れた研削力を発揮することが認められた。

囚みにこの結果は大昭第3裏に示したジルコニア正方品結局の比率に対応しているものであって、 添加量 O . O 5 Wt % のものでは従来のものとあまり大差はなく、10 Wt % を超えると立方品の折出に伴い従来のものよりダウンする傾向がみられる。

しかしながら酸化イットリウムの、添加は、全体としては研制力の増大傾向を若しくするものであり、しかもこのテスト結果がわずか10分間の研制時間における比較であることを考えたとき、実際の使用にあっては極めて優れた研削性能の向上に寄与し得るものである。

实施例2.

低粒中のジルコニアが 4 0 % となるように配合: 計算を行ない、パイヤー法アルミナ (99.6% Al 2 O 1 ) 5 0 kg 、 ジルコニア (96% Zr Oz ) 3 2 . 6 kg に、ジルコニアに対し値 位パーセントで 2 . 5 %に相当する酸化チクン (9 5 % T l Oz ) O . 8 kg を混合し、さらに 酸化イットリウムを含む緑土類鉱物(第 1 数に示 す分析値のもの)を前3 種の全盤に対し 1 、 2 . 5 、5 wt % 添加して、電気炉において 9 5 V 、 3 0 0 kw で溶融し、しかる後急冷間化して各種 添加量についての溶融鋳造物を得た。

これらの妨盗物の分析値を第5数に示す。なお 試料番号1は前記実施例1と同じく酸化イットリウム無磁加の従来のものである。

(以下次頁)

1	2		Ø	5 <sup>†</sup> &	•			
計劃	Y2 O) を ( 含むほ士類 鉢物の返加則 (V1%)	A1203	Zr Oz	TI Oz	. भृः	F0¿O3	Si O	Na z O
1	0	56,94	41,61	1,20	· <b>-</b>	0,05	0,12	0.08
9	1	57.36	40,97	1,04	0.42	0,05	0,13	0,03
10	2,5	56,56	41_04	1.09	1,05	0.06	0, 16	0,04
11	5	56, 19	40.33	1,12	2.11	0,05	0,17	0.03

ヰ Y₂ O₂ その他婦土類

次にこの得られた好造物をインペラーブレーカー及びロールクラッシャーを用いて繰り返し粉砕を行ない、JISR-6001に定められた粒度#24、#60で各々採収した。

粒度#24についてXQ額所によるジルコニアの結晶形の比率を第6表に示す。

(以下次頁)

1	2	01 6 & B	4
क्षांत्रक्ष	正方為Zr Oz (%)	MMAZrOz (%)	/ 紅方瓜Zr Oz (%)
1	30.1	69,9	0
9	96,4	3, 6	0
10	100,0	0	0
11	100.0	0	0

#### 特周昭59-227726(6)

又線回折の結果明らかなように、酸化イットリウムを含む私土類鉱物を添加すれば、酸化イットトリウムの添加の場合と同じく、添加しないのにカーチタニア系配粒(試力の低力)に比し、正方島の結局が著しく増大する傾向ではることが認められ、2.5~5 vi %の添加量ではもっていることが認められる。

次に粒度#60について研削性能の試験を行なった。

試験条件は実施例1と同様である。その結果については第7表に示す。

$\frac{1}{r}$	第7表 · 2	_			
試料循号	黑松	M	削	£3 (	g }
1	94.4	(	R	atio	100)
9	118,7	(		n	126)
10	120.0	(		#	127)
11	119.2	(		*	126)

また第3図における山壌(b) はこの第7数に おける実積研削量値をプロットしたグラフである。

上記第7表あるいは第3図から明らかなように、 酸化イットリウム添加の実施例1の場合と同様に、 酸化イットリウムを含む稀土類鉱物を添加する共 品低粒においても優れた研削力を発揮することが みとめられた。

#### 实施例3.

表に示す。

低粒中のジルコニアが40%になるように配合計算を行ない、バイヤー法アルミナ50 kg . ジルコニア32.6 kn にジルコニアに対して近日パーセントで5、15 wt %の酸化チタン1.7、4.9 kg を混合し、酸化イットリウムを約3種の混合物に対し近母パーセントで0.5 wt % 添加したものについて特造物の分析値、粒度#24についてのX線回折によるジルコニア結晶形の比率及び粒度#60についての別位研例量を翻定した。その結果をそれぞれ第8裏、第9表及び第10

なお比較のため、酸化チタン2. 5 91%の添加

量のもの、及びそれぞれのチタニア添加肌のもの についての酸化イットリウム無添加の場合の値も 併記した。

、 また試験方法はいずれも実施例1及び実施例2 と同様である。

(以下次页)

#### 特問昭59-227726(プ)

l	N	17		<b>វា</b>	8 &				
-	1	1	**					,	
<b>新月</b>	Ti O. Esined (%)	(MW) KMMII (MW)		Zr O.	Ti O	Y2 O3	Fe <sub>z</sub> O <sub>3</sub>	SI O,	Na <sub>*</sub> O
ī	2.5	0	5G, 94	41,61	1.20	-	0.05	0.12	0.08
4	2,5	0,5	57.65	40,85	1.02	0.27	0.03	0,14	0.04
12	5	0	57.06	40,63	2,06		0.03	0.18	0.04
13	5	0,5	5G,78	40.71	2.04	0.25	0.04	0.15	0,03
14	15	0	54,16	39.72	5.91		0.03	0,14	0.04
15	15	0.5	53,41	40,24	15,83	0_29	0.04	0.15	0.04

		•	104-030 621168(7)
	,\	2 第9数	5
	試料指导	正方為 Z r O z	# M B Z r O z
		(%)	(%)
٠	1	3,0.1	69.9
	4	87.9	12.1
	12	33.0	67.0
	13	90.2	9.8
	14	34.2	65.8
	15	93.6	6.4

, \	新10数 , 2			
就和番号	深档	मा है	14 <u>51</u> (	g )
1	94.4	( [	Ratio	100)
4	115.7	(	"	123)
12	95.0	(	n	100)
13	112.5	(		119)
14	92.0	•	<i>n</i>	97)
15	117.3	(	#	124)

上記表からも明らかなように、酸化チタンの添加冊の均別に従い正方島 Z ト O 2 の残存率は増大傾向を示しているが、それにも増して酸化イットリウムの添加による著しい増大傾向が認められた。またこれに対応して研削力の増大傾向が深積研削量の数値結果から認められる。
実施例 4 .

3 O、 4 O % の酸化チタン(9 5 % T i O z )を 添加し、さらに酸化イットリウムを前 3 紙の物質 全盘に対して O . 5 wt % 添加したものをそれぞれ、 電気炉を用いてアーク熱で溶融・急冷切化し、これを一般的な砥粒の生産方式に従って発粒し、 J I S # 1 2 の砥粒とした。

これらのもの、すなわち酸化イットリウム組織加のジルコニア25%タイプ及び32%タイプのあのと、酸化イットリウム添加のジルコニア25%タイプのものについて抗酸的性制定試験を行なった。抗酸降性制定には単粒圧増強度を用いた。この方法は、試料を1680~2000ミクロンに整粒して、輸分法により小試料とし、その中からランダムに100個採取して、これを2トンアムスラー圧縮器で1個ずつ耐圧強度を測定して、その平均値を単粒圧増強度とした。

これらの砥粒の単粒圧慢強度を第11表に、III 粒圧壊強度とTi Oz / Zr Oz との関係を第4 図に示す。

#### 特問昭59-227726(8)

第4回より明らかな様に、いずれのタイプにおいても下i O 2 / Z r O 2 が単量パーセントで10~20%の範囲内で、max 飲を示し、40%になると低下するが、特に酸化イットリウムを添加したものについては、Z r O 2 25%タイプにあっては勿論、32%タイプと比較しても更にすぐれた抗破砕性を発揮することが認められた。

従って 距 研 閉 の 中 で も 特 に そ の 軽 研 削 に 対 し 、 ま た さ ら に チ タ ン 合 金 に 対 し て 格 別 に 良 好 に 適 応

D 11 €										
क्षाध	月タイプ		Ti Oz /Zr Oz (%)							
		0	2.5	5	10	15	20	30	40	
16	Y: O: 無透加 Zr O: 25% タイプ	•	48.8	51,9	61.0	60.0	58.9	52.2	46,7	
17	Y2 O2 知道加 Z1 O2 32% タイプ 〇	46,3	47,5	53,8	83.0	63.3	62.0	49.3	46.0	
18	Yz O3 知惑的 Zr Oz 25% タイプ	48, 1	_	55.8	64,7	64.2	63, 3	54,1	4B.0	

して使用できるものである。

また比較的ジルコニア員の少ないアルミナージ ルコニア共晶体を含む研削材にあっても従来に比 較すればその抗破砕性等の特性において一段と向 上し得たものである。

#### 4. 図面の簡単な説明

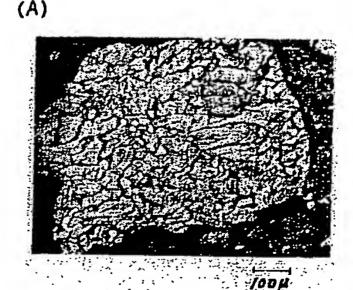
第1回(A)(B)は従来の酸化イットリウム 無添加のアルミナージルコニア共島混合物(試料 番号1)の拡大写真及びその模式図、

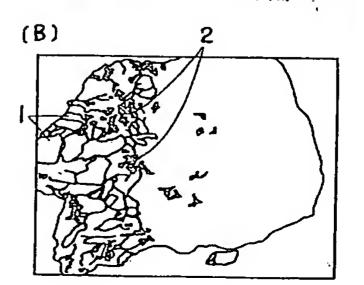
第2図(A)(B)はこの発明の一実施例である酸化イットリウム蒸加のアルミナージルコニア 共品混合物(試料番号3)の拡大写真及びその模式図、

第3回は同次施例及び他次施例である肢化イットリウム添加の研削材及び酸化イットリウムを含む紙上粉鉱物添加の研削材における酸化イットリウム等の添加量と祭稿研削量の関係図、

第4回はジルコニア量が比較的少ない研削材に おける砂化チタンの抵加量と単粒圧度強度との関 係図である。

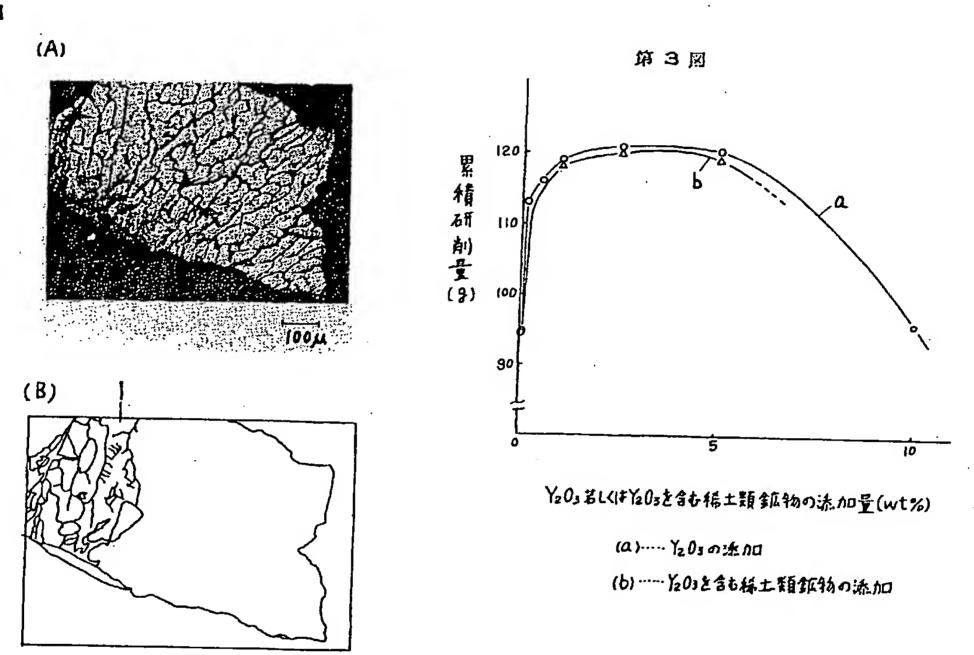
熔 1 図



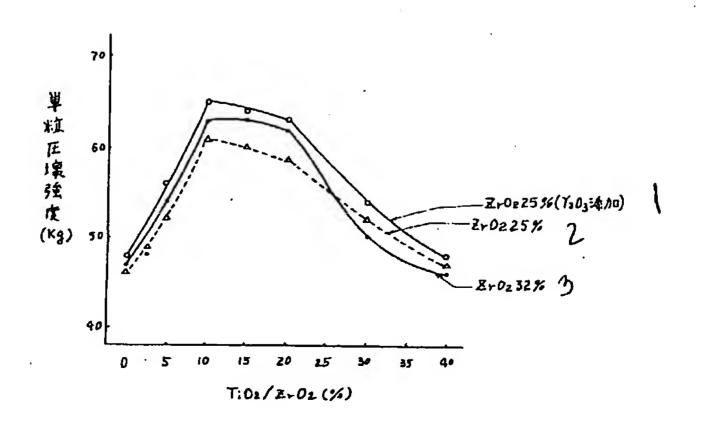


### 特別昭59-227726(9)

第2図



第 4 図



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